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Solid Oxide Cell and Stack Testing, Safety and Quality Assurance (SOCTESQA)

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In the EU-funded project "SOCTESQA" partners from Europe and Singapore are working together to develop uniform and industry wide test procedures and protocols for solid oxide cells and stacks (SOC) cell/stack assembly. New application fields which are based on the operation of the SOC cell/stack assembly in the fuel cell (SOFC), in the electrolysis (SOEC) and in the combined SOFC/SOEC mode are addressed. This covers the wide field of power generation systems, e.g. stationary SOFC µ-CHP, mobile SOFC APU and SOFC/SOEC power-to-gas systems. This paper presents the results which have been achieved so far. Besides a summary of existing test procedures a so called "test matrix" was created. This document includes generic test modules, e.g. current-voltage curves, electrochemical impedance spectroscopy, thermal cycling, electrical current cycling and long-term tests both under steady-state and dynamic operating conditions. The application specific test programs are created by combining several of these test modules.

Motivation

Up to now the development of solid oxide cell/stack assembly units has proceeded to cover a wide range of applications. Solid oxide cells (SOCs) are applied for all operation modes like solid oxide fuel cell (SOFC), solid oxide electrolysis cell (SOEC) and combined SOFC/SOEC as well as steady-state and dynamic operations. The headings "power-to-gas" (1) and "power-to-gas-to-power" (2) explain the development of SOEC and combined SOFC/SOEC in the field of renewable energies as energy conversion systems. In fact SOFCs are actually commercially available as micro-combined heat and power generators (µCHP) e.g. from companies like Hexis AG (Switzerland) (3) or Ceramic Fuel Cells Ltd. (Australia) (4). In dynamic operation SOFCs are used as auxiliary power units (APU) to create electricity in trucks (5). So far a lot of research has been done. However, the successful market launch requires also reliable assessment, testing and prediction of performance and durability. Also the complexity of high temperature solid oxide assembly units and the appendant test systems increases the need of detailed test schemes, procedures and protocols. Figure 1 shows these complex correlations which should be considered to get reliable object test results.

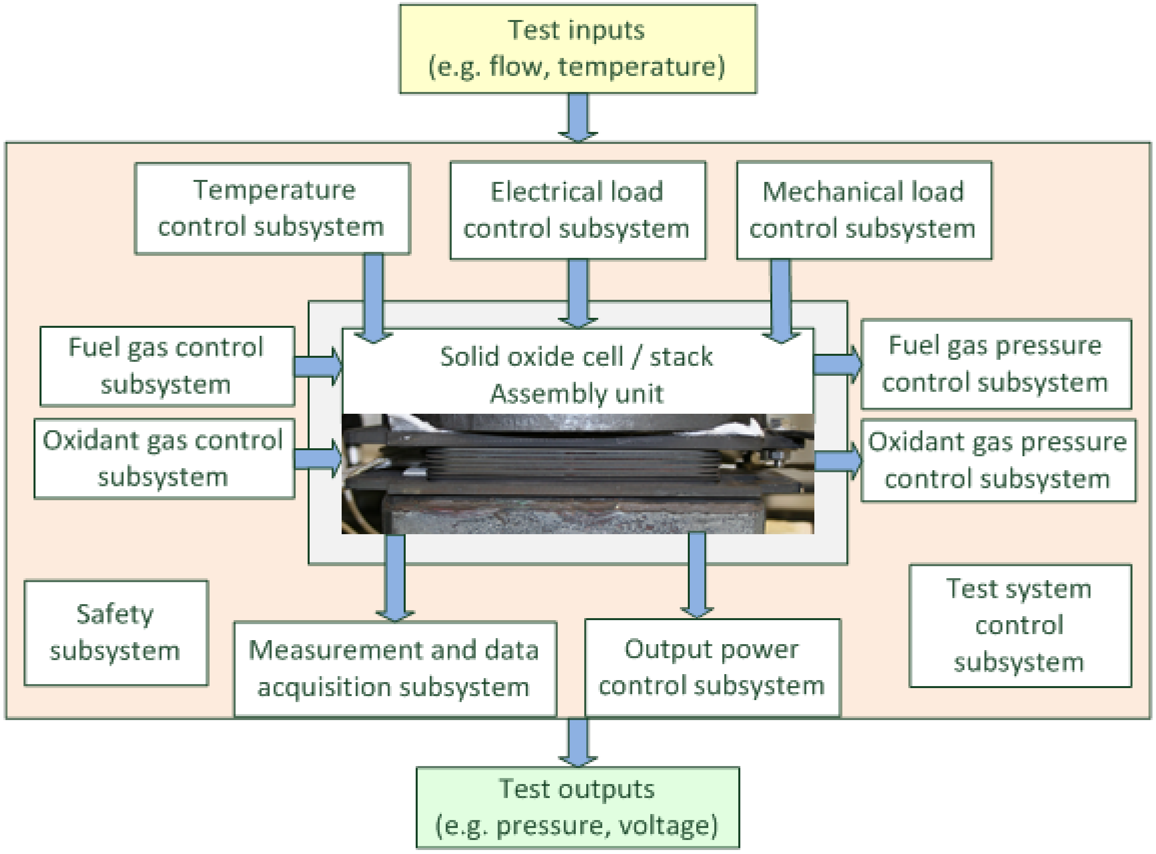

Figure 1: Schematic graph of a test system for high temperature solid oxide assembly unit

Test matrix and literature survey

With the aim to develop uniform test procedures the definition of an overall test matrix for SOFC, SOEC and combined SOFC/SOEC operations and the survey of existent SOC test procedures have been the first steps in the "SOCTESQA" project.

Definition of an overall test matrix

The test matrix includes an overview of possible tests, so called test modules (TMs), covering the whole application range of SOFC, SOEC and combined SOFC/SOEC operations. Later on these test modules can be combined to generate test programs in order to realize application-oriented testing. In order to reveal and consider the industries needs the industrial advisory board (IAB) with members of important cell and stack suppliers and also producers of components and assemblies has been involved from the very beginning. Relevant technical details and specifications have been determined with the help of application specific fact sheets. With the input from industry, the results of previous projects (6), (7) and the great experience of the "SOCTESQA" project partners

(DLR, CEA, DTU, ENEA, JRC, EIFER, NTU) 18 possible test modules could be identified. The combination of these generic test modules with the relevance for the different applications leads to the application-oriented test matrix which is shown in Table I. The four major applications identified so far are stationary SOFC micro-combined heat and power generation (SOFC-µCHP), mobile SOFC auxiliary power units used in cars or trucks (SOFC-APU), SOEC H_2 production (power-to-gas) and combined SOFC/SOEC electricity storage (power-to-gas-to-power). The relevance of each test module for these four applications is indicated in the test matrix. The "x" shows the relevance for the dedicated application, the symbol "-" means that the test module is irrelevant in this case.

Table I: Test Matrix

Generic Test Modules		**Applications**			
		Stationary **SOFC-µCHP**	**Mobile** **SOFC-APU**	**SOEC for H_2 production** ***power-to-gas***	**Combined SOFC/SOEC for electricity storage** ***power-to-gas-to-power***
TM01	Leakage test	x	x	x	x
TM02	Start-up	x	x	x	x
TM03	Current-voltage characteristics	x	x	x	x
TM04	Electrochemical impedance spectroscopy	x	x	x	x
TM05	Current interruption	x	x	x	x
TM06	Cyclic voltammetry	-	-	-	-
TM07	Reactant utilisation	x	x	x	x
TM08	Reactant gas composition	x	x	-	-
TM09	Temperature sensitivity	x	x	x	x
TM10	Pressure sensitivity	-	-	x	x
TM11	Mechanical load sensitivity	x	x	x	x
TM12	Operation under constant current	x	x	x	x
TM13	Operation under varying current	x	x	x	x
TM14	Thermal cycling	x	x	x	x
TM15	Redox cycling	x	x	x	x
TM16	Shut-down	x	x	x	x
TM17	Vibration test	-	x	-	-
TM18	Emergency Stop	x	x	x	x

Additionally to the operating phase which includes the application specific test modules, also test modules for start-up and shut-down procedures are being developed.

Gas tightness was defined as one of the most important requirements for an SOC cell or even more for an SOC stack. Therefore the first test module addresses leakage tests followed by TM02 describing the start-up procedures with the note that start-up parameters always depend on the applied cell/stack design.

The test modules for the operating phase can be divided into two parts. Standard operations like “Current-voltage characteristics” (TM03), “Electrochemical impedance spectroscopy” (TM04) or “Operation under constant current” (TM12) are essential and independent of different applications. The test matrix showed that most of the defined test modules are more or less suitable for all of the identified applications. Only some few test modules like “Reactant gas composition” (TM08) or “Pressure sensitivity” (TM10) are application specific and relevant either for SOFC or SOEC and combined SOFC/SOEC operations. Moreover procedures for “Shut-down” (TM16) and also “Emergency stops” (TM18) are defined.

An important task of the “SOCTESQA” project is that all basic test modules will be established and validated among the partners by different experimental validation phases. At the end of the project the reviews will result in modified test modules confirmed by round robin tests.

Survey of existing SOC test procedures

In order to reach the aim of uniform test procedures it is necessary to specify important terms, definitions and nomenclatures. This assumes not only to focus on SOC specified items but also on fuel and electrolysis cell development in general to simplify matters and benefit from any experiences. Therefore this survey includes besides previous projects, e.g. “FCTESTNET” (8), “FCTESQA” (PEMFC, SOFC, MCFC) (9), “METSOFC” (10), “REALSOFC 600”, “SOFC-LIFE”, “SCOTAS-SOFC”, “METSAPP”, “ADEL” and “RELHY” and on-going European projects like, “STACK-TEST” (PEMFC) (11) also procedures exterior to the mentioned projects (12), (13), (14), (15), (16). Especially in the case of definitions and terminology the documents published by the International Electrotechnical Commission (IEC) (17), (18) have been considered. The results of the literature survey are represented in Figure 2. Available and relevant documents are assigned to the test modules defined in the test matrix. Documents concerning neither SOFC nor SOEC are defined as “others”, e.g. low temperature fuel cells.

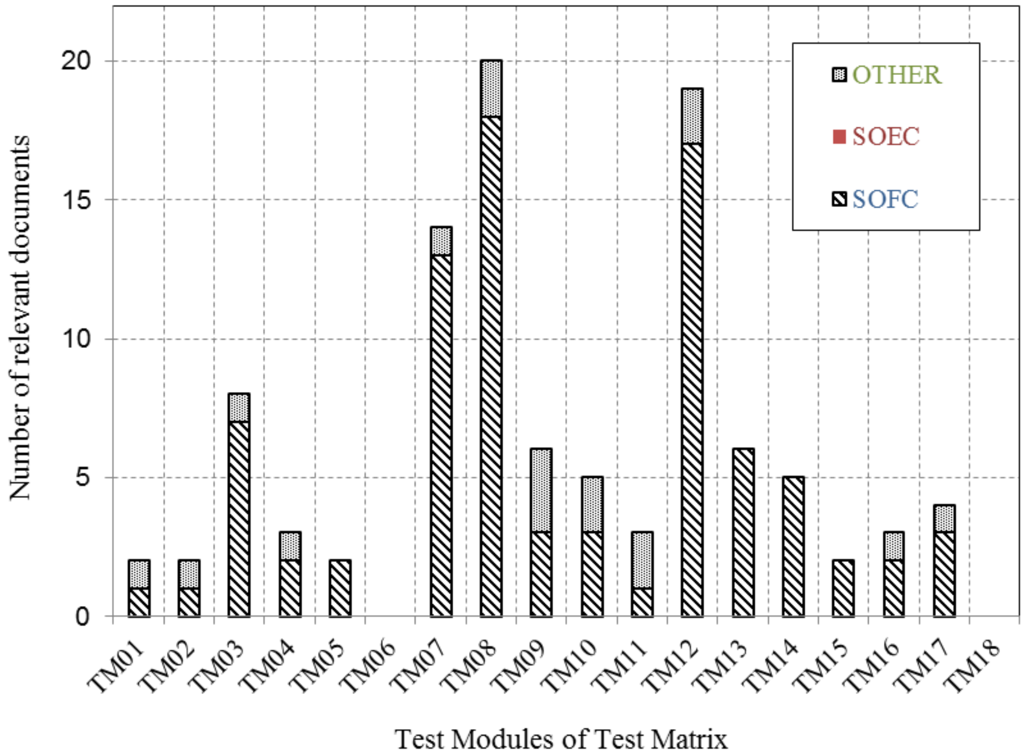

Figure 2: Correlation between existing and relevant documents and defined test modules

Figure 2 shows that there are only a few specifications of test procedures for SOCs in general. Most of the above mentioned projects concentrate on SOFC with main focus on single cell tests and system level tests under steady-state conditions. The survey also shows an obvious gap concerning specifications of test procedures for SOEC operation. One reason may be the small number of projects which deal with this item. In addition a lot of project results are confidential and therefore not open to the public. For this reason documents e.g. from “RELHY” and “ADEL” projects which deal with SOEC cannot be considered.

Concerning the single test modules a proportional large number of documents for “Reactant utilisation” (TM07), “Reactant gas composition” (TM08) and “Operation under constant current” (TM12) are available. Other test modules like “Cyclic voltammetry” (TM06) or even “Electrochemical impedance spectroscopy” are hardly represented.

In summary the literature review shows once more the urgent need of application specific testing procedures addressing function, performance, durability and degradation of SOC cells/stacks.

Interface between test object and test system

Just as significant as the specification of important terms, and nomenclatures is the definition of the interface between test object and test system according to Figure 1. In this context test input parameters (TIPs) and test output parameters (TOPs) also have to be defined.

Using the example of an SOC stack as test object, Figure 3 shows the correlation between test object, test environment, test input parameters and test output parameters. The test object is exposed to test inputs or operating conditions and delivers test outputs or object test results. In the "SOCTESQA" project test input parameters are defined as parameters whose values can be set in order to define the test operating conditions of the cell/stack. TIPs have to be controllable and measurable. On the other hand test output parameters are parameters that indicate the performance of the cell/stack as a function of TIPs. Variation of TIPs leads to a response of the cell/stack indicated by the change of TOPs. TOPs do not have to be controllable but must be measurable.

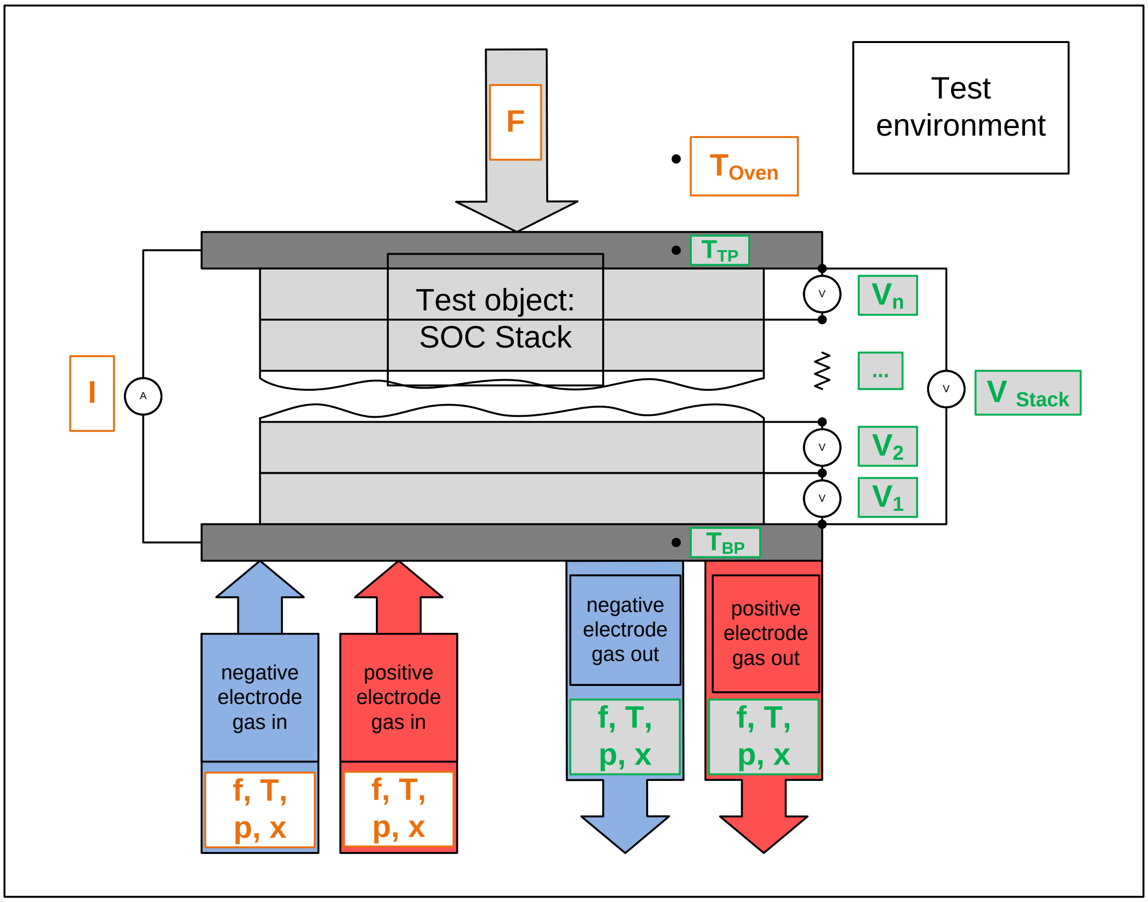

Figure 3: Interfaces between SOC stack test object and test environment

The physical cell/stack interfaces from the test environment (test station) to the SOC cell/stack test object can be separated in media, electric, heat and mechanical interfaces. Moreover, these interfaces can be classified according to their input or output nature, each containing one or several input or output parameters, respectively. Media interfaces are the reactants inlet and outlet. Electric interfaces are the load and power supply and voltage wiring. Heat interface is the oven and the mechanical interface considers the mounting of the cell/stack assembly unit on the test station which may supply compression force to the cell/stack. The mentioned interfaces are defined by different physical parameters. For the reactants at cell/stack inlet and/or outlet these parameters are temperature (T), flow (mass or volume, f), pressure (p) and gas composition (x). The electric interface parameters are cell/stack current (I), stack voltage (V_{stack}) and the voltages of the cell or the individual repeating units (V_{cell} or $V_{RU,i}$). Oven temperature

(T_{oven}), stack top plate temperature (T_{TP}), stack bottom plate temperature (T_{BP}) and cell or stack internal temperature (T_{cell} or $T_{stack,intern}$) belong to the heat interface parameters. The cell or stack compression force (F_{compr}) is the mechanical interface parameter. Ideally, the positions for the setting/ monitoring of the input/output parameters are as close as possible to the boundary of the test object and the test environment.

Test program

The "SOCTESQA" project defines test programs that have different objectives and scope. The test programs are comprised of test modules, in which the actual test parameters, specific conditions and test setup are described in details. The test program describes the objective and scope of the test as well as the test operating conditions (TOC), which is typically where the general application specific parameters are given, such as reactant composition, operating temperature etc. Generally a test program comprises a start-up and shut-down module to safely start-up the cell/stack and to document the initial and final performance of the SOC cell/stack. Each test program may contain several test modules for characterization, e.g. current-voltage curves, electrochemical impedance spectroscopy (EIS), cyclic voltammetry and for performance and durability testing, with e.g. dynamic operating profiles or long-term stationary operation. The selected test modules depend on the objective and are geared to the application. The test program specifies also the order of test modules, the number and in some instances also parameters such as total test time, e.g. in the case of durability testing where intermittent characterization throughout the test is needed. It is possible to run more than one test program in sequence, whereby time and stress from start-up and shut-down in test programs can be minimized. The relationship between test programs and modules in general is illustrated in Figure 4.

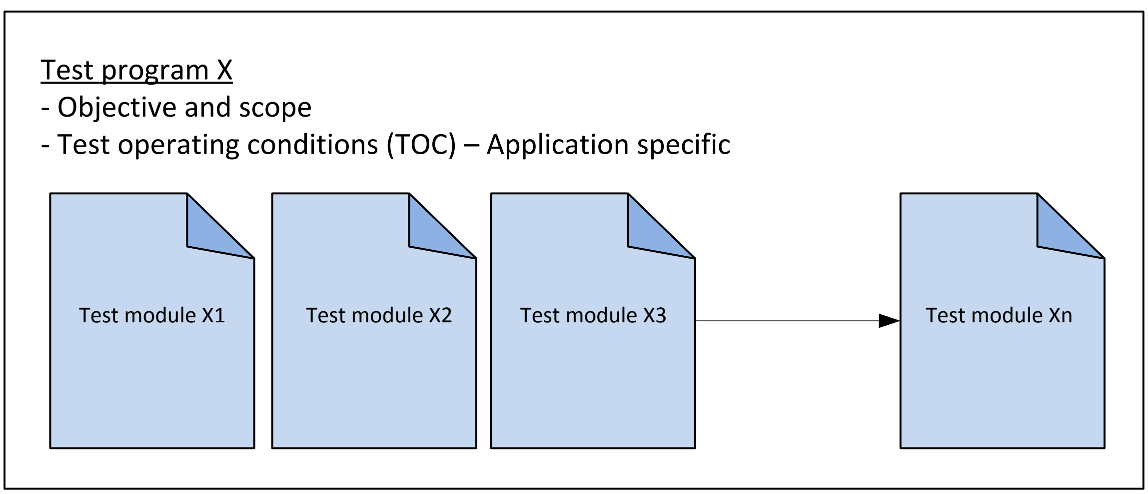

Figure 4: Illustration of relationship between test programs and modules

For better understanding Figure 5 shows a possible test program for durability testing as an example. It consists of a series of different test modules for characterization and performance evaluation, each module with its own specific objective as well as the test program objective. The initial current-voltage curve in conjunction with the electrochemical impedance spectroscopy attends to document the initial performance. The comparison with the final current-voltage measurements leads to the degradation rate of the cell/stack. To identify the performance-limiting factor as well as the degradation mechanism the electrochemical impedance spectroscopy is a useful tool. The applied test module for current-voltage characteristics (TM03) defines the key parameters for measuring a current-voltage curve such as the current variation rate, gas flow rates, gas temperatures as well as the way to present the data. The procedure to obtain the electrochemical impedance diagram and the way to present the data are described in test module 04. Many applications dictate a dynamic operation of the stack, varying load with time. Therefore test module 13 “Operation under varying current” includes typical load profiles for each application to simulate the real-life operation modes. The major output will be the degradation rate of the cell/stack.

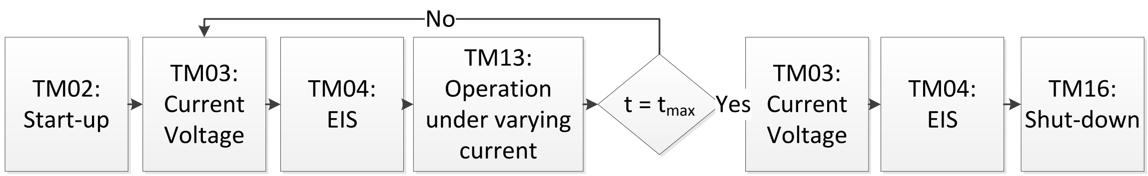

Figure 5: Example of a test program structure, comprising several test modules for characterization and performance evaluation

By using this test programs comparable and reliable results will be obtained. This is the great benefit of the “SOCTESQA”-project.

Outlook

The validation of the SOC specific test modules and programs will be performed by an initial test station comparison followed by two validation and review loops. Therefore the next step in the “SOCTESQA”-project will be the test station validation. In order to avoid differences in performance and behavior caused by the test station itself, the interfaces between test samples and test stations have to be adapted. Also the differences of the test stations among the project partners, different kind of fuel gases or other effects have to be taken into account and harmonized if necessary. For the validation of the test programs, stacks with high reproducibility and established by the same stack supplier are chosen. In this way influences resulted from the stacks can be minimized. For the initial test station validation firstly some selected test modules are applied. The results of the different testing partners will be compared. Possible deviations will be evaluated by parameter sensitivity analysis. Afterwards the test modules will be reviewed and modified based on the experimental validation.

Acknowledgments

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